

## **Analytical Laboratory**

13339 Hagers Ferry Road Huntersville, NC 28078-7929 McGuire Nuclear Complex - MG03A2 Phone: 980-875-5245 Fax: 980-875-4349

## **Order Summary Report**

Order Number:	J13080184				
Project Name:	WWTS - Biweekly				
Customer Name(s):	Robbin Jolly, Bill Kennedy				
Customer Address:	253 Plant Allen Road				
	Belmont, NC 28012				
Lab Contact:	Jason C Perkins	Phone:	980-875-5348		
Report Authorized By: (Signature)		Dat	te:	8/23/2013	
(Oignature)	Jason C Perkins				

#### **Program Comments:**

Please contact the Program Manager (Jason C Perkins) with any questions regarding this report.

#### **Data Flags & Calculations:**

Any analytical tests or individual analytes within a test flagged with a Qualifier indicate a deviation from the method quality system or quality control requirement. The qualifier description is found at the end of the Certificate of Analysis (sample results) under the qualifiers heading. All results are reported on a dry weight basis unless otherwise noted. Subcontracted data included on the Duke Certificate of Analysis is to be used as information only. Certified vendor results can be found in the subcontracted lab final report. Duke Energy Analytical Laboratory subcontracts analyses to other vendor laboratories that have been qualified by Duke Energy to perform these analyses except where noted.

#### Data Package:

This data package includes analytical results that are applicable only to the samples described in this narrative. An estimation of the uncertainty of measurement for the results in the report is available upon request. This report shall not be reproduced, except in full, without the written consent of the Analytical Laboratory. Please contact the Analytical laboratory with any questions. The order of individual sections within this report is as follows:

Job Summary Report, Sample Identification, Technical Validation of Data Package, Analytical Laboratory Certificate of Analysis, Analytical Laboratory QC Reports, Sub-contracted Laboratory Results, Customer Specific Data Sheets, Reports & Documentation, Customer Database Entries, Test Case Narratives, Chain of Custody (COC)

#### Certification:

The Analytical Laboratory holds the following State Certifications: North Carolina (DENR) Certificate #248, South Carolina (DHEC) Laboratory ID # 99005. Contact the Analytical Laboratory for definitive information about the certification status of specific methods.

## Sample ID's & Descriptions:

Page 2 of 16

Sample ID	Plant/Station	Collection Date and Time	Collected By	Sample Description
2013018951	ALLEN	07-Aug-13 7:13 AM	CRAIG McHUGH	FGD Purge Eff
2013018952	ALLEN	07-Aug-13 7:18 AM	CRAIG McHUGH	EQ Tank Eff
2013018953	ALLEN	07-Aug-13 7:21 AM	CRAIG McHUGH	BioReactor 1 Inf
2013018954	ALLEN	07-Aug-13 7:26 AM	CRAIG McHUGH	BioReactor 2 Inf
2013018955	ALLEN	07-Aug-13 7:24 AM	CRAIG McHUGH	BioReactor 2 Eff
2013018956	ALLEN	07-Aug-13 8:02 AM	CRAIG McHUGH	Filter Blk
2013018957	ALLEN	23-Jul-13 3:00 PM	C. KNOX	TRIP BLANK
7 Total Samples				

## **Technical Validation Review**

## **Checklist:**

COC and .pdf report are in agreement with sample totals and analyses (compliance programs and procedures).

All Results are less than the laboratory reporting limits. ☐ Yes ✓ No

All laboratory QA/QC requirements are acceptable. ✓ Yes ☐ No

## **Report Sections Included:**

✓ Job Summ	nary Report		✓ Sub-contracted Laboratory Results
✓ Sample Id	lentification		☐ Customer Specific Data Sheets, Reports, & Documentation
<b>✓</b> Technical	Validation of Data Package		Customer Database Entries
Analytical	Laboratory Certificate of Analysis		✓ Chain of Custody
☐ Analytical	Laboratory QC Report		☐ Electronic Data Deliverable (EDD) Sent Separately
Reviewed By:	DBA Account	Date:	8/23/2013

V\_AS&C

# **Certificate of Laboratory Analysis**

This report shall not be reproduced, except in full.

#### Order # J13080184

Site: FGD Purge Eff Sample #: 2013018951

Collection Date: 07-Aug-13 7:13 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
NITRITE + NITRATE (COLORIME	TRIC)							
Nitrite + Nitrate (Colorimetric)	21	mg-N/L		0.25	25	EPA 353.2	08/19/2013 12:36	BGN9034
INORGANIC IONS BY IC								
Bromide	67	mg/L		5	50	EPA 300.0	08/13/2013 21:09	JAHERMA
MERCURY (COLD VAPOR) IN W	ATER							
Mercury (Hg)	67.0	ug/L		2.5	50	EPA 245.1	08/16/2013 08:21	DKJOHN2
TOTAL RECOVERABLE METALS	S BY ICP							
Boron (B)	94.2	mg/L		0.5	10	EPA 200.7	08/14/2013 11:10	MHH7131
` ,	•	Ü						
DISSOLVED METALS BY ICP-MS Selenium (Se)	<u>5</u> 119	ug/L		10	10	EPA 200.8	08/19/2013 10:33	DJSULL1
` ,		ug/L		10	10	LI A 200.0	00/13/2013 10.33	DJJOLLI
TOTAL RECOVERABLE METALS	S BY ICP-MS							
Arsenic (As)	204	ug/L		10	10	EPA 200.8	08/16/2013 13:59	DJSULL1
Cadmium (Cd)	< 10	ug/L		10	10	EPA 200.8	08/16/2013 13:59	DJSULL1
Chromium (Cr)	232	ug/L		10	10	EPA 200.8	08/16/2013 13:59	DJSULL1
Copper (Cu)	262	ug/L		10	10	EPA 200.8	08/16/2013 13:59	DJSULL1
Nickel (Ni)	252	ug/L		10	10	EPA 200.8	08/16/2013 13:59	DJSULL1
Selenium (Se)	1350	ug/L		10	10	EPA 200.8	08/16/2013 13:59	DJSULL1
Silver (Ag)	< 10	ug/L		10	10	EPA 200.8	08/16/2013 13:59	DJSULL1
Zinc (Zn)	281	ug/L		10	10	EPA 200.8	08/16/2013 13:59	DJSULL1
SELENIUM SPECIATION - (Analy								

Complete

Vendor Parameter

Site: EQ Tank Eff Sample #: 2013018952

Collection Date: 07-Aug-13 7:18 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
MERCURY (COLD VAPOR) IN WATE	<u>:R</u>							
Mercury (Hg)	32.7	ug/L		2.5	50	EPA 245.1	08/16/2013 08:23	DKJOHN2
TOTAL RECOVERABLE METALS BY	( ICP							
Boron (B)	69.2	mg/L		0.5	10	EPA 200.7	08/14/2013 11:14	MHH7131
DISSOLVED METALS BY ICP-MS								
Selenium (Se)	56.3	ug/L		10	10	EPA 200.8	08/19/2013 10:36	DJSULL1

Vendor Method

## **Certificate of Laboratory Analysis**

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#### Order # J13080184

Site: EQ Tank Eff Sample #: 2013018952

Collection Date: 07-Aug-13 7:18 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
TOTAL RECOVERABLE METALS BY	ICP-MS							
Arsenic (As)	103	ug/L		10	10	EPA 200.8	08/16/2013 14:03	DJSULL1
Cadmium (Cd)	< 10	ug/L		10	10	EPA 200.8	08/16/2013 14:03	DJSULL1
Chromium (Cr)	112	ug/L		10	10	EPA 200.8	08/16/2013 14:03	DJSULL1
Copper (Cu)	138	ug/L		10	10	EPA 200.8	08/16/2013 14:03	DJSULL1
Nickel (Ni)	150	ug/L		10	10	EPA 200.8	08/16/2013 14:03	DJSULL1
Selenium (Se)	761	ug/L		10	10	EPA 200.8	08/16/2013 14:03	DJSULL1
Silver (Ag)	< 10	ug/L		10	10	EPA 200.8	08/16/2013 14:03	DJSULL1
Zinc (Zn)	149	ug/L		10	10	EPA 200.8	08/16/2013 14:03	DJSULL1

Site: BioReactor 1 Inf Sample #: 2013018953

Collection Date: 07-Aug-13 7:21 AM Matrix: OTHER

SELENIUM SPECIATION - (Analysis Performed by Applied Speciation and Consulting, LLC)

Complete

Vendor Parameter

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
NITRITE + NITRATE (COLORIME	TRIC)							
Nitrite + Nitrate (Colorimetric)	8.7	mg-N/L		0.25	25	EPA 353.2	08/19/2013 12:37	BGN9034
Mercury by EPA 200.8 - (Analysis	s Performed by	Applied Sp	eciation and	Consulti	ina. LLC)			
Vendor Parameter	Complete	ug/l	ooiation and	o o no anti-	<u></u>	Vendor Method		V AS&C
	·	3						
TOTAL RECOVERABLE METALS	BY ICP							
Boron (B)	56.0	mg/L		0.5	10	EPA 200.7	08/14/2013 11:18	MHH7131
DISSOLVED METALS BY ICP-MS	<u> </u>							
Selenium (Se)	61.6	ug/L		10	10	EPA 200.8	08/19/2013 10:40	DJSULL1
TOTAL RECOVERABLE METALS	BY ICP-MS							
Arsenic (As)	< 10	ug/L		10	10	EPA 200.8	08/16/2013 14:06	DJSULL1
Cadmium (Cd)	< 10	ug/L		10	10	EPA 200.8	08/16/2013 14:06	DJSULL1
Chromium (Cr)	< 10	ug/L		10	10	EPA 200.8	08/16/2013 14:06	DJSULL1
Copper (Cu)	18.0	ug/L		10	10	EPA 200.8	08/16/2013 14:06	DJSULL1
Nickel (Ni)	25.4	ug/L		10	10	EPA 200.8	08/16/2013 14:06	DJSULL1
Selenium (Se)	55.9	ug/L		10	10	EPA 200.8	08/16/2013 14:06	DJSULL1
Silver (Ag)	< 10	ug/L		10	10	EPA 200.8	08/16/2013 14:06	DJSULL1
Zinc (Zn)	< 10	ug/L		10	10	EPA 200.8	08/16/2013 14:06	DJSULL1

Vendor Method

V\_AS&C

2013018954

## **Certificate of Laboratory Analysis**

This report shall not be reproduced, except in full.

### Order # J13080184

Site: BioReactor 2 Inf Sample #:

Collection Date: 07-Aug-13 7:26 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
Mercury by EPA 200.8 - (Analysis	Performed by A	pplied Sp	eciation and	Consult	ing, LLC)			
Vendor Parameter	Complete	ug/l				Vendor Method		V_AS&C
TOTAL RECOVERABLE METALS	BY ICP							
Boron (B)	53.5	mg/L		0.5	10	EPA 200.7	08/14/2013 11:22	MHH7131
TOTAL RECOVERABLE METALS	BY ICP-MS							
Arsenic (As)	< 10	ug/L		10	10	EPA 200.8	08/16/2013 14:10	DJSULL1
Cadmium (Cd)	< 10	ug/L		10	10	EPA 200.8	08/16/2013 14:10	DJSULL1
Chromium (Cr)	< 10	ug/L		10	10	EPA 200.8	08/16/2013 14:10	DJSULL1
Copper (Cu)	< 10	ug/L		10	10	EPA 200.8	08/16/2013 14:10	DJSULL1
Nickel (Ni)	< 10	ug/L		10	10	EPA 200.8	08/16/2013 14:10	DJSULL1
Selenium (Se)	14.0	ug/L		10	10	EPA 200.8	08/16/2013 14:10	DJSULL1
Silver (Ag)	< 10	ug/L		10	10	EPA 200.8	08/16/2013 14:10	DJSULL1
Zinc (Zn)	< 10	ug/L		10	10	EPA 200.8	08/16/2013 14:10	DJSULL1

Site: BioReactor 2 Eff Sample #: 2013018955

Collection Date: 07-Aug-13 7:24 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
NITRITE + NITRATE (COLORIMET	RIC)							
Nitrite + Nitrate (Colorimetric)	< 0.01	mg-N/L		0.01	1	EPA 353.2	08/19/2013 12:38	BGN9034
INORGANIC IONS BY IC								
Bromide	56	mg/L		5	50	EPA 300.0	08/13/2013 21:28	JAHERMA
Mercury by EPA 200.8 - (Analysis	Performed by A	Applied Sp	eciation and	Consulti	ng, LLC)			
Vendor Parameter	Complete	ug/l				Vendor Method		V_AS&C
TOTAL RECOVERABLE METALS	BY ICP							
Boron (B)	55.5	mg/L		0.5	10	EPA 200.7	08/14/2013 11:27	MHH7131
TOTAL RECOVERABLE METALS	BY ICP-MS							
Arsenic (As)	< 5	ug/L		5	5	EPA 200.8	08/16/2013 14:13	DJSULL1
Cadmium (Cd)	< 5	ug/L		5	5	EPA 200.8	08/16/2013 14:13	DJSULL1
Chromium (Cr)	< 5	ug/L		5	5	EPA 200.8	08/16/2013 14:13	DJSULL1
Copper (Cu)	< 5	ug/L		5	5	EPA 200.8	08/16/2013 14:13	DJSULL1
Nickel (Ni)	< 5	ug/L		5	5	EPA 200.8	08/16/2013 14:13	DJSULL1
Selenium (Se)	< 5	ug/L		5	5	EPA 200.8	08/16/2013 14:13	DJSULL1
Silver (Ag)	< 5	ug/L		5	5	EPA 200.8	08/16/2013 14:13	DJSULL1
Zinc (Zn)	< 5	ug/L		5	5	EPA 200.8	08/16/2013 14:13	DJSULL1

## **Certificate of Laboratory Analysis**

This report shall not be reproduced, except in full.

#### Order # J13080184

Site: BioReactor 2 Eff Sample #: 2013018955

Collection Date: 07-Aug-13 7:24 AM Matrix: OTHER

Analyte Result Units Qualifiers RDL DF Method Analysis Date/Time Analyst

SELENIUM SPECIATION - (Analysis Performed by Applied Speciation and Consulting, LLC)

Vendor Parameter Complete Vendor Method V\_AS&C

**TOTAL DISSOLVED SOLIDS** 

TDS **8600** mg/L 25 1 SM2540C 08/20/2013 15:30 DSBAKE1

Site: Filter Blk Sample #: 2013018956

Collection Date: 07-Aug-13 8:02 AM Matrix: OTHER

Analyte Result Units Qualifiers RDL DF Method Analysis Date/Time Analyst

DISSOLVED METALS BY ICP-MS

Selenium (Se) <1 ug/L 1 1 EPA 200.8 08/19/2013 10:15 DJSULL1

Site: TRIP BLANK Sample #: 2013018957

Collection Date: 23-Jul-13 3:00 PM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
TOTAL RECOVERABLE METALS BY	<u>ICP</u>							
Boron (B)	< 0.05	mg/L		0.05	1	EPA 200.7	08/14/2013 11:06	MHH7131
TOTAL RECOVERABLE METALS BY	ICP-MS							
Arsenic (As)	< 1	ug/L		1	1	EPA 200.8	08/16/2013 13:56	DJSULL1
Cadmium (Cd)	< 1	ug/L		1	1	EPA 200.8	08/16/2013 13:56	DJSULL1
Chromium (Cr)	< 1	ug/L		1	1	EPA 200.8	08/16/2013 13:56	DJSULL1
Copper (Cu)	< 1	ug/L		1	1	EPA 200.8	08/16/2013 13:56	DJSULL1
Nickel (Ni)	< 1	ug/L		1	1	EPA 200.8	08/16/2013 13:56	DJSULL1
Selenium (Se)	< 1	ug/L		1	1	EPA 200.8	08/16/2013 13:56	DJSULL1
Silver (Ag)	< 1	ug/L		1	1	EPA 200.8	08/16/2013 13:56	DJSULL1
Zinc (Zn)	< 1	ug/L		1	1	EPA 200.8	08/16/2013 13:56	DJSULL1



18804 Northcreek Parkway Bothell, WA, 98011 Tel: (425) 483-3300 Fax: (425) 483-9818 www.appliedspeciation.com

August 21, 2013

Jay Perkins Duke Energy Analytical Laboratory Mail Code MGO3A2 (Building 7405) 13339 Hagers Ferry Rd. Huntersville, NC 28078 (704) 875-5245

Project: Allen – FGD WWTS (Bi-Monthly Routine) (LIMS# J13080184)

Mr. Perkins,

Attached is the report associated with four (4) aqueous samples submitted for total mercury and selenium speciation analysis on August 8, 2013. The samples were received in a sealed cooler at -0.2°C on August 9, 2013. Selenium speciation analysis was performed via ion chromatography inductively coupled plasma collision reaction cell mass spectrometry (IC-ICP-CRC-MS). Mercury quantitation was performed via cold vapor inductively coupled plasma mass spectrometry (CV-ICP-MS). Any issues associated with the analysis are addressed in the following report.

If you have any questions, please feel free to contact me at your convenience.

Sincerely,

Russell Gerads Vice President

Applied Speciation and Consulting, LLC

#### Applied Speciation and Consulting, LLC

Report prepared for:

Jay Perkins
Duke Energy Analytical Laboratory
Mail Code MGO3A2 (Building 7405)
13339 Hagers Ferry Rd.
Huntersville, NC 28078

Project: Allen – FGD WWTS (Bi-Monthly Routine) (LIMS# J13080184)

August 21, 2013

#### 1. Sample Reception

Three (3) aqueous samples were submitted for selenium speciation analysis on August 8, 2013. Three (3) additional samples were submitted for total mercury quantitation. All samples were received in acceptable condition on August 9, 2013 in a sealed container at -0.2°C.

All samples were received in a laminar flow clean hood, void of trace metals contamination and ultra-violet radiation, and were designated discrete sample identifiers. The 40mL borosilicate glass vial submitted for total mercury was preserved with bromine monochloride (BrCl) solution. The resulting sample was stored in a secure polyethylene container, known to be free from trace metals contamination, until the analyses could be performed.

An aliquot of each sample requiring selenium speciation evaluation was filtered  $(0.45\mu m)$  and each filtrate was stored in a secure, monitored cryofreezer (maintained at a temperature of -80°C) until selenium speciation analysis could be performed via ion chromatography inductively coupled plasma collision reaction cell mass spectrometry (IC-ICP-CRC-MS).

#### 2. Sample Preparation

All sample preparation is performed in laminar flow clean hoods known to be free from trace metals contamination. All applied water for dilutions and sample preservatives are monitored for contamination to account for any biases associated with the sample results.

<u>Total Mercury Quantitation by CV-ICP-MS</u> All samples and preparation blanks for total mercury quantitation were preserved with 2% (v/v) BrCl. The resulting samples were analyzed for mercury via cold vapor inductively coupled plasma mass spectrometry (CV-ICP-MS).

<u>Selenium Speciation Analysis by IC-ICP-CRC-MS</u> Prior to analysis, an aliquot of each sample was filtered with a syringe filter (0.45µm) and injected directly into a sealed autosampler vial. No further sample preparation was performed as any chemical alteration of a sample may shift the equilibrium of the system, resulting in changes in speciation ratios.

#### 3. Sample Analysis

All sample analysis is preceded by a minimum of a five-point calibration curve spanning the entire concentration range of interest. Calibration curves are performed at the beginning of each analytical day. All calibration curves, associated with each species of interest, are standardized by linear regression resulting in a response factor. All sample results are **instrument blank corrected** to account for any operational biases associated with the analytical platform.

Prior to sample analysis, all calibration curves are verified using second source standards which are identified as initial calibration verification standards (ICV).

Ongoing instrument performance is identified by the analysis of continuing calibration verification standards (CCV) and continuing calibration blanks (CCB) at a minimum interval of every ten analytical runs.

<u>Total Mercury Quantitation by CV-ICP-MS</u> The sample fractions for total mercury quantitation were analyzed by cold vapor inductively coupled plasma mass spectrometry (CV-ICP-MS) on August 16, 2013. Aliquots of each sample are reacted with a reductant inline and transported to a gas-liquid separator. The volatile elemental mercury that is formed is then swept by a stream of argon gas into a radio frequency (RF) plasma where energy-transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially-pumped vacuum interface and separated on the basis of their mass-to-charge ratio (m/z) by a mass spectrometer. A solid-state detector detects ions transmitted through the mass analyzer and the resulting current is processed by a data handling system.

<u>Selenium Speciation Analysis by IC-ICP-CRC-MS</u> Each sample for selenium speciation analysis was analyzed by ion chromatography inductively coupled plasma collision reaction cell mass spectrometry (IC-ICP-CRC-MS) on August 9, 2013. An aliquot of each sample is injected onto an anion exchange column and mobilized by a basic (pH > 7) gradient. The eluting selenium species are then introduced into a radio frequency (RF) plasma where energy-transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially-pumped vacuum interface and travel through a pressurized chamber (CRC) containing a reaction gas which preferentially reacts with interfering ions of the same target mass to charge ratios (m/z). A solid-state detector detects ions transmitted through the mass analyzer and the resulting current is processed by a data handling system.

Retention times for each eluting species are compared to known standards for species identification.

#### 4. Analytical Issues

The overall analyses went well and no significant analytical issues were encountered. All quality control parameters associated with these samples were within acceptance limits.

The estimated method detection limits (eMDLs) for selenite, selenate, and selenocyanate are generated from replicate analyses of the lowest standard in the calibration curve. Not all selenium species are present in preparation blanks; therefore, eMDL calculations based on preparation blanks are artificially biased low.

The eMDL for methylseleninic acid and selenomethionine is calculated from the average eMDL of selenite, selenate, and selenocyanate. The calibration does not contain methylseleninic acid or selenomethionine due to impurities in these standards which would bias the results for other selenium species.

The eMDL for mercury has been calculated using the standard deviation of the preparation blanks preserved and analyzed concurrently with the submitted samples.

If you have any questions or concerns regarding this report, please feel free to contact me.

Sincerely,

Russell Gerads

Vice President

Applied Speciation and Consulting, LLC

### Total Mercury & Selenium Speciation Results for Duke Energy Project Name: Allen - FGD WWTS (Bi-Monthly Routine) Contact: Jay Perkins LIMS #J13080184

Date: August 21, 2013
Report Generated by: Russell Gerads
Applied Speciation and Consulting, LLC

#### Sample Results

							Unknown Se
Sample ID	Total Hg	Se(IV)	Se(VI)	SeCN	MeSe(IV)	SeMe	Species (n)
FGD Purge Eff	NR	74.1	31.9	ND (<2.0)	8.9	ND (<2.0)	0 (0)
BioReactor 1 Inf	0.6740	11.9	27.2	ND (<0.49)	1.15	ND (<0.49)	0.68 (1)
BioReactor 2 Inf	0.0463	NR	NR	NR	NR	NR	NR
BioReactor 2 Eff	0.0156	ND (<0.93)	ND (<0.059)	ND (<0.49)	ND (<0.49)	ND (<0.49)	0 (0)

All results reflect the applied dilution and are reported in µg/L

NR = Analysis not requested

ND = Not detected at the applied dilution

SeCN = Selenocyanate

MeSe(IV) = Methylseleninic acid

SeMe = Selenomethionine

Unknown Se Species = Total concentration of all unknown Se species observed by IC-ICP-MS

n = number of unknown Se species observed

### Total Mercury & Selenium Speciation Results for Duke Energy Project Name: Allen - FGD WWTS (Bi-Monthly Routine) Contact: Jay Perkins LIMS #J13080184

Date: August 21, 2013 Report Generated by: Russell Gerads Applied Speciation and Consulting, LLC

### **Quality Control Summary - Preparation Blank Summary**

Analyte (µg/L)	PBW1	PBW2	PBW3	PBW4	Mean	StdDev	eMDL*	eMDL 5x	eMDL 250x	eMDL 1000x
Hg	-0.0004	0.0001	-0.0006	-0.0005	-0.0004	0.0003	0.0002	0.0009	-	-
Se(IV)	0.025	0.020	0.016	0.012	0.018	0.005	0.004	-	0.93	3.7
Se(VI)	0.000	0.012	0.035	0.000	0.012	0.016	0.000	-	0.059	0.24
SeCN	0.000	0.000	0.000	0.000	0.000	0.000	0.002	-	0.49	2.0
MeSe(IV)	0.000	0.000	0.000	0.000	0.000	0.000	0.002	-	0.49	2.0
SeMe	0.000	0.000	0.000	0.000	0.000	0.000	0.002	-	0.49	2.0

eMDL = Estimated Method Detection Limit

### **Quality Control Summary - Certified Reference Materials**

Analyte (µg/L)	CRM	True Value	Result	Recovery	
Hg	NIST 1641d	1568	1647	105.0	
Se(IV)	LCS	4.79	4.92	102.8	
Se(VI)	LCS	4.74	4.70	99.2	
SeCN	LCS	4.46	4.53	101.6	
MeSe(IV)	LCS	3.24	3.24	100.2	
SeMe	LCS	4.66	4.49	96.5	

<sup>\*</sup>Please see narrative regarding eMDL calculations

### Total Mercury & Selenium Speciation Results for Duke Energy Project Name: Allen - FGD WWTS (Bi-Monthly Routine) Contact: Jay Perkins LIMS #J13080184

Date: August 21, 2013 Report Generated by: Russell Gerads Applied Speciation and Consulting, LLC

#### **Quality Control Summary - Matrix Duplicates**

Analyte (µg/L)	Sample ID	Rep 1	Rep 2	Mean	RPD
Hg	BioReactor 2 Eff	0.0156	0.0168	0.0162	7.4
Se(IV)	Batch QC	ND (<3.7)	ND (<2.0)	NC	NC
Se(VI)	Batch QC	617.4	609.4	613.4	1.3
SeCN	Batch QC	ND (<2.0)	ND (<2.0)	NC	NC
MeSe(IV)	Batch QC	ND (<2.0)	ND (<2.0)	NC	NC
SeMe	Batch QC	ND (<2.0)	ND (<2.0)	NC	NC

ND = Not detected at the applied dilution

NC = Value was not calculated due to one or more concentrations below the eMDL

### **Quality Control Summary - Matrix Spike/ Matrix Spike Duplicate**

Analyte (µg/L)	Sample ID	Spike Conc	MS Result	Recovery	Spike Conc	MSD Result	Recovery	RPD
Hg	BioReactor 2 Eff	2.000	2.085	103.4	2.000	2.084	103.4	0.0
Se(IV)	Batch QC	5560	5609	100.9	5560	5553	99.9	1.0
Se(VI)	Batch QC	5045	5343	93.7	5045	5370	94.3	0.5
SeCN	Batch QC	4575	3996	87.4	4575	3983	87.1	0.3

Page 15 of 16 Customer must Complete 2) Client: 1301895 BlOper, Unit: LAB USE ONLY Refinquished By T DUKE ENERGY. Relicquished By Seafil or ked by Respondshed. Customer to complete appropriate columns to right Se Speciation Bottle WWTS (Bi-Monthly Routine) AS00 20003 Robbin Jolly, Bill Kennedy omer to sign & date below - fill out from left to right 8 Allen - FGD 9)Res. Type: **Duke Energy Analytical Laboratory** <sup>13</sup>Sample Description or ID Mail Code MGO3A2 (Building 7405) **BMCEFGD** 13339 Hagers Ferry Rd Huntersville, N. C. 28078 (704) 875-5245 Fax: (704) 875-4349 BioReactor 2 Eff BioReactor 2 Inf BioReactor 1 Inf FGD Purge Eff DatorTone EQ Tank Eff. Metals Trip Blk Filter BK 4)Fax No: Marie 10)Resp. Center: [2]Seal/Lock Opened By 8-7-13 3-7-13 8-7-13 8-7-13 0724 8-7-13 0726 Seal/Lock Opened By 8-7-13 0802 Crais MAY appropriate non-shaded areas. 1-13/1500 Date Sampling conducted: 2nd and 4th Monday Carry Customer to complete all AS&C PO#650910 6721 07/8 0713 O Crain Milaria Crais M/de/ Capacot Crain Milly (3) MATRIX OTHER 1°Presery.:1=HCL 2=H,SO, 3=HNO なら 5=Node Filtering of soluble Se performed in the field <sup>7</sup>Comp. 16Analyses Required <sup>18</sup>Grab TDS Water PROGRAM Return kit to Ray Lidke, @ Allen Br (Dionex) RCRA Waste 3,4 \* Metals\* + Hg 245.1 ‡ Ť į 3,4 Se, soluble (no dig.) Ground Supplies (
Supp 2,4 Customer, IMPORTANT! NO3-NO2 Please indicate desired turnaround. Hg 200.8 (V\_AS&C) \_ <sup>22</sup>Requested Turnaround 21 Days 7 Days Ĉ E COPY to CLIENT ORIGINAL to LAB, <sup>19</sup>Page 1 of 2 DISTRIBUTION ·48 H: \* Add. Cost Will Apply Se, speciation - vendor to ASSC (important to piace filled back into both baggles)

As CH. C. CL. N. Se Ad. Zn by TRIMINES

B by TRAVICE

1\*\*=No Hg analyzed

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CHAIN OF CUSTODY RECORD AND ANALYSIS REQUEST FORM

#### CHAIN OF CUSTODY RECORD AND ANALYSIS REQUEST FORM **Duke Energy Analytical Laboratory Analytical Laboratory Use Only** Page 16 of 46 DISTRIBUTION MATRIX OTHER NC X Mail Code MGO3A2 (Building 7405) J13080184 13339 Hagers Ferry Rd ORIGINAL to LAB, Huntersville, N. C. 28078 Logged By Date & Time SAMPLE PROGRAM COPY to CLIENT Ground (704) 875-5245 8/8/13 958 D. Baker NPDES Fax: (704) 875-4349 5°C UST 2)Phone No: Allen - FGD RCRA Waste **WWTS (Bi-Monthly Routine)** Cooler Temp (C) AS&C Preserv.:1=HCL 4)Fax No: 2=H2SO4 3=HNO3 2) Client: Robbin Jolly, Bill Kennedy PO#650910 4 3,4 3,4 2,4 5=None MR# Se, speciation - vendor to AS&C (Important to place filled bottle back into both baggies) 16 Analyses Required Mail Code: 6)Process: **BMCEFGD** 5)Business Unit: 20003 dig Hg 200.8 (V\_AS&C) Metals\* + Hg 245.1 10)Resp. Center: 9)Res. Type: Customer to complete all 8)Oper. Unit: 01) **AS00** appropriate non-shaded areas. Br (Dionex) Se, soluble N03-N02 Sampling conducted: 2nd and 4th Monday LAB USE ONLY Se Speciation Bottle TDS <sup>13</sup>Sample Description or ID Signature Time 1 Crais Milder FGD Purge Eff 0718 Crais Meldry 1 EQ Tank Eff. 62 1 1 BioReactor 1 Inf Cray Wylder 8-7-13 0726 1 BioReactor 2 Inf · 54 1 1 BioReactor 2 Eff 11 55 8-7-13 0802 Crais M/14/ Filter Blk 7-23 1500 Cobnor Metals Trip Blk 1 51-57 Filtering of soluble Se performed in the field Return kit to Ray Lidke, @ Allen Customer to sign & date below - fill out from left to righ Date/Time 2) Accepted By <sup>22</sup>Requested Turnaround desired turnaround. 1) Relinquished By Date/Time Date/Time 3) Relinquished By Date/Time 6)Accepted By: Date/Time 5)Relinquished By Date/Time 8)Accepted By: Date/Time \*48 Hr 7) Relinquished By stomer, dicate Date/Time 10) Seal/Lock Opened By Date/Time 9)Seal/Locked By \* Add. Cost Will Apply Date/Time 12)Seal/Lock Opened By 3 11)Seal/Locked By Ple Comments \* Metals=As, Cd, Cr, Cu, Ni, Se, Ag, Zn by TRM/IMS, B by TRM/ICP 1\*\*=No Hg analyzed